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13. ABSTRACT *(Maximum 200 words)*

This project focused on the study of the effects of microstructural features on the fracture process of room and high temperature structural ceramic materials that operate under cyclic loading environments. The ability to model these effects and the prediction of the damage behavior also become an important consideration, as it provides a direct link to the design cycle for both, processing and design engineers.

The project evaluated cyclic effects, high cycle fatigue behavior, and elevated temperature effects under these conditions. Evaluations of the effects of cyclic loading are performed using pre-cracked tensile test specimens (PFT). It is observed that hysteretic load-displacement loops arise as the primary characteristic of the behavior, although gross-slip behavior is seen under certain circumstances.

The results on this study conclude that the most important parameters responsible for the observed behavior, whether hysteretic of gross residual displacement behavior, are contact point and unbroken ligament densities. The results show a direct link between the behavior observed and the microstructural features that are active; mainly elastic bridges and that dissipate frictional energy. It is also concluded that a distribution of residual stresses and grain stiffness is required to correlate with the observed behaviors. Evidence of both, large and small scale debris is also found after cyclic loading. Large-scale debris is primarily found in small grain size aluminas, whereas small-scale debris observed in high cyclic fatigue specimens. Small-scale debris is primarily the result of mechanical wear.

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FRACTURE PROCESS ZONE MODELING OF SMALL CRACKS IN STRUCTURAL CERAMICS UNDER STATIC AND CYCLIC LOADING

AFOSR Grant: F49620-98-1-0429 Final Report, August 2001

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Abstract

This project focused on the study of the effects of microstructural features on the fracture process of room and high temperature structural ceramic materials that operate under cyclic loading environments. The ability to model these effects and the prediction of the damage behavior also become an important consideration, as it provides a direct link to the design cycle for both, processing and design engineers.

The project evaluated cyclic effects, high cycle fatigue behavior, and elevated temperature effects under these conditions. Evaluation of the effects of cyclic loading are performed using pre-cracked tensile test specimens (PFT). It is observed that hysteretic load-displacement loops arise as the primary characteristic of the behavior, although gross-slip behavior is seen under certain circumstances.

The results of this study conclude that the most important parameters responsible for the observed behavior, whether hysteretic or gross residual displacement behavior, are contact point and unbroken ligament densities. The results show a direct link between the behavior observed and the microstructural features that are active, mainly elastic bridges and the conversion from the elastic to sliding bridges that dissipate frictional energy. It is

also concluded that a distribution of residual stresses and grain stiffness is required to correlate with the observed behaviors. Evidence of both, large and small scale debris is also found after cyclic loading. Large scale debris is primarily found in small grain size aluminas, whereas small scale debris is observed in high cyclic fatigue specimens. Small scale debris is primarily the result of mechanical wear.

1. Objectives: No change from proposal

2. Basic Research Issues

- Determination of crack growth toughening mechanisms in structural ceramics at all temperature conditions of relevance.
- Relate operative toughening mechanisms to the microstructure in a universal manner.
- Measurement methods of specimen displacements at high temperatures were developed and adapted to the demands of the present challenge.
- Development of a descriptive model of the physical principles.

3. Approach

- PFT methods and SEM analysis of the crack path will serve as the primary tools for the determination of the link between the microstructure and the behavior.
- Micromechanical modeling is utilized to examine the role of the microstructure on the observed cyclic behavior.

4. Significant Results

Fracture Process Zone Characterization: Cyclic Loading Results

The single most prominent feature of the load-displacement behavior in a PFT specimen under cyclic loading is the formation of a hysteresis loop. A typical set of load-displacement curves is shown in Figure 1. The maximum test load was progressively increased for each cycle. Although the data curves would be normally displaced by the residual amount of the previous cycle, all the curves are shown at the origin. The data is shown this way to validate the assumption that no gross wake zone damage is occurring during the initial stages of the test, as indicated by the unchanged specimen compliance. A linear-elastic behavior is observed as the specimen is loaded to 0.22 kg which is then unloaded. Following the behavior relating to the role of contact density we may conclude that the majority of the contact points in this portion of the wake zone act as elastic contacts throughout the load range of cycle 1. This is a reasonable assumption, as the smallest COD characterizes this part of the wake zone, which is immediately behind the crack tip. One would expect to find the greatest population bridging grains in this region.

With the increase of the maximum load in cycles 2 through 4 in Figure 1, a hysteresis loop develops. This is an indication that some of the elastic points present at lower load levels begin to slide, thus converting to slip contact points. The tangential shear stress acting on these contact points has increased sufficiently to overcome the critical shear stress, which results in the increasing compliance observed.

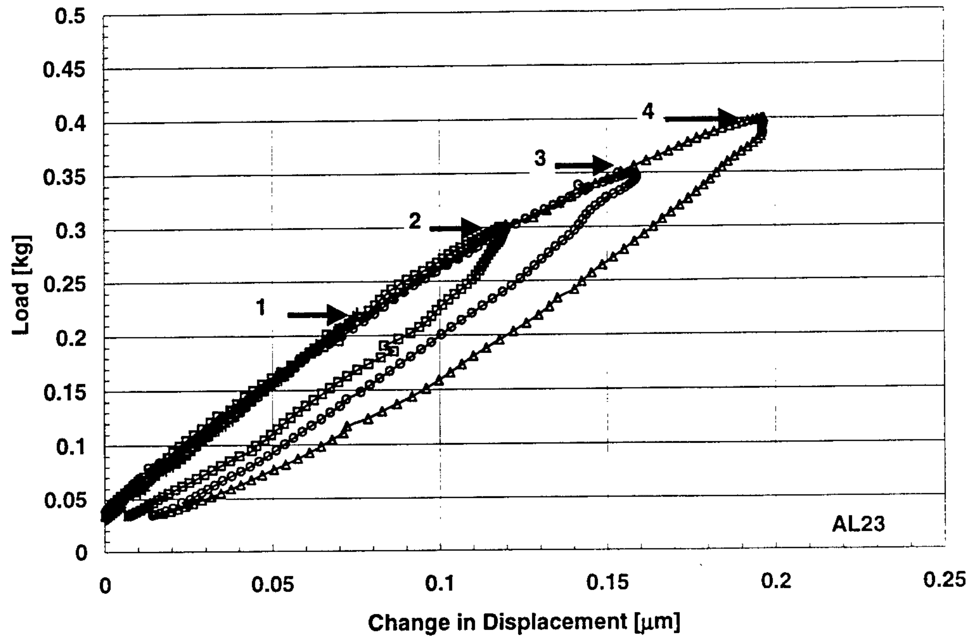


Figure 1 Effect of progressive loading in the evolution on a nearly closed hysteresis loops in alumina

Because the nature of the loading and unloading event includes the dissipation of energy by frictional sliding, it is observed that the hysteresis loops do not fully close when unloaded. The nature of the PFT test, where only tension loading is allowed due to the nature of the geometry of the lower loading groove dictates that the energy available to return the loop to a nearly closed state comes solely from the elastic energy stored in the system (elastic contact points and unbroken ligaments).

A distinctively different behavior, namely open loop behavior, was observed for all of PFT specimens with larger initial CODs, regardless of the microstructure. A typical open loop behavior is presented in Figure 2. In this test, the PFT specimen is initially loaded to 0.15 kg, where nearly linear elastic behavior is observed. Further cycles at larger maximum loads, result in a hysteresis loop characterized by a sharp transition point and a large residual displacement. This behavior is associated with the formation of a new bridging system and generation of large-scale debris.

The case presented here is different from the case presented before, in that any subsequent loading cycle that is applied to this specimen results in a distinctively open loop behavior. The formation of open loop behavior is the result of gross slip, and the continuous formation of new bridging systems with subsequent loading. At this load, a mass conversion of stick to slip contact points occurs leading to the gross slip behavior observed in cycles 2, and 3. Continuation of this test at the level of loading that causes gross slip would have resulted in certain failure of the specimen in the cycles immediately following the ones shown. Instead of continuing the cycling at the same loading conditions, the maximum load is reduced back to the initial load of 0.15 kg, to assess the condition of the wake zone. It is observed that a nearly closed hysteresis loop is formed once again, but closer examination of the loop, by comparison to the initial test cycle reveals degradation of the wake zone, as indicated by the increased specimen compliance.

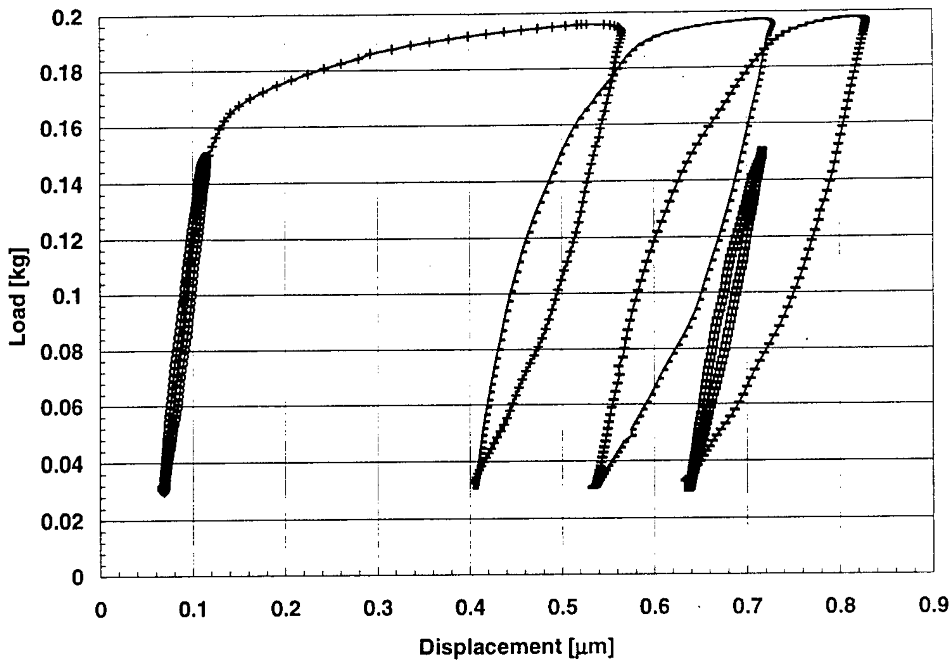


Figure 2 Effect of increased loading and resulting open loop behavior on a PFT 3 AL23 specimen

The typical effects of long term, or high, cyclic loading are shown in Figure 3. Cycles 1, 100, 1000 and 4000 are presented for the same PFT. Here, it is observed that as load cycling progresses, the frictional sliding force (force that resists the movement of slip contacts) is reduced. This may result from a combination of two different mechanisms. Firstly, the frictional coefficient between contact points may be reduced due to some smoothing of the interface. Secondly, removal of interface material, as a result of wear, would make the normal forces on the bridging grains to decrease. Both of these mechanisms acting alone or simultaneously could be responsible for the observed behavior. Also, it is of note that the load which determines the onset of sliding, i.e. where the initial loading compliance begins to increase, reduces with increasing cycle number. The onset of sliding is shown for both cycles 1 and 4000. Interestingly, the initial compliance remains constant, which indicates that the number of contact points does not change throughout this test.

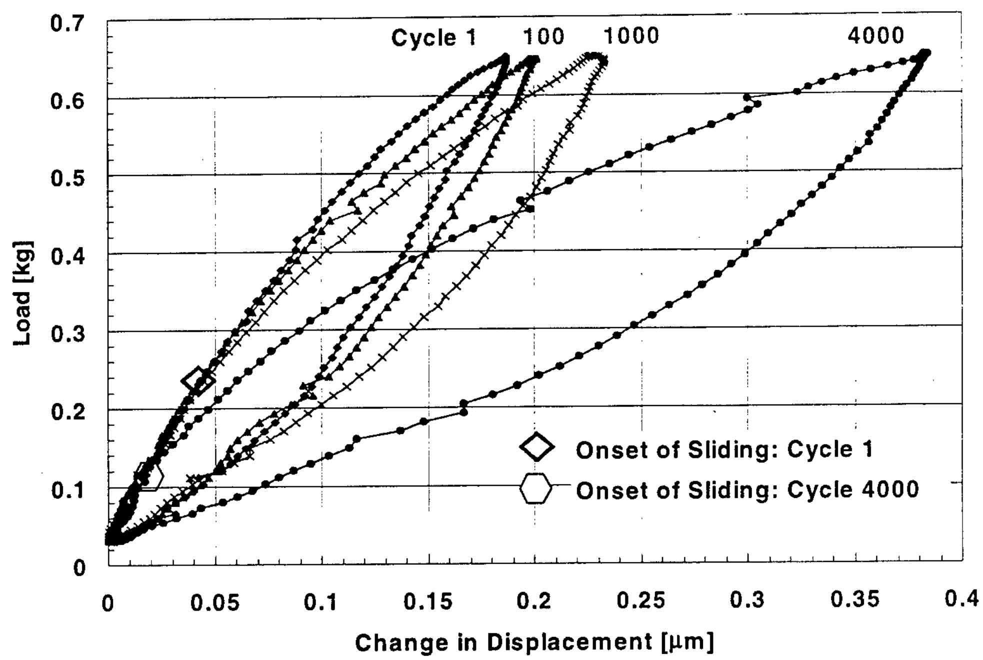
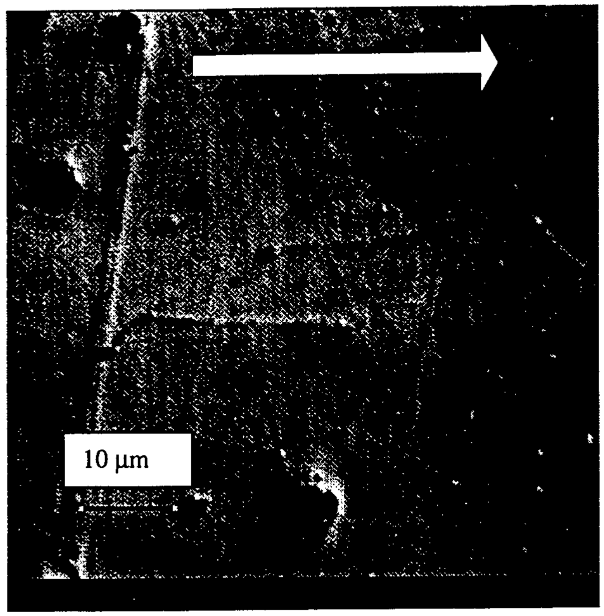


Figure 3 Effects of Long Term Cyclic Loading

SEM observation of crack path: Bridging by unbroken ligaments

An understanding of how bridging elements are set up is a key in any attempt to tailor the microstructure aiming to producing tougher ceramics. In order to understand the nature and the behavior of the bridging ligaments under certain loading conditions, SEM (Scanning Electron Microscopy) crack path studies were conducted on Double Cantilever Beam (DCB) specimens. It was found that the bridging near the crack tip is promoted by unbroken ligaments. Two different SEM micrographs of the crack path in alumina are shown on Figure 4. They identify discontinuities (Crack Jumping) of the crack path near the crack tip. These features are found to extend to a distance of about 1.5 to 2 mm. Furthermore, it appeared from the micrographs of the polished-etched surfaces that the crack had propagated predominantly in an intergranular mode, although some transgranularly fractured grains were also observed.

In the near crack tip region (estimated to be of 1.5 to 2 mm in length), unbroken ligaments, which bridged the crack, were mainly present and we believe that they promote rising R-curves through closure forces. Our own observations gave no indication of dispersed microcracking. Thus, it is worth mentioning at this point that the lack of experimental evidence for a microcracked zone that is associated with the primary crack excludes microcracking as the mechanism for producing a rising R-curve. Depending on their shape, We believe that the discontinuities represent a first stage of bridge formation and certainly will transform into frictional bridges after further loading. We exclude here the fact that the coplanar discontinuities (Figure 4.B) develop into frictional bridges unless the crack deflects from its horizontal plane.



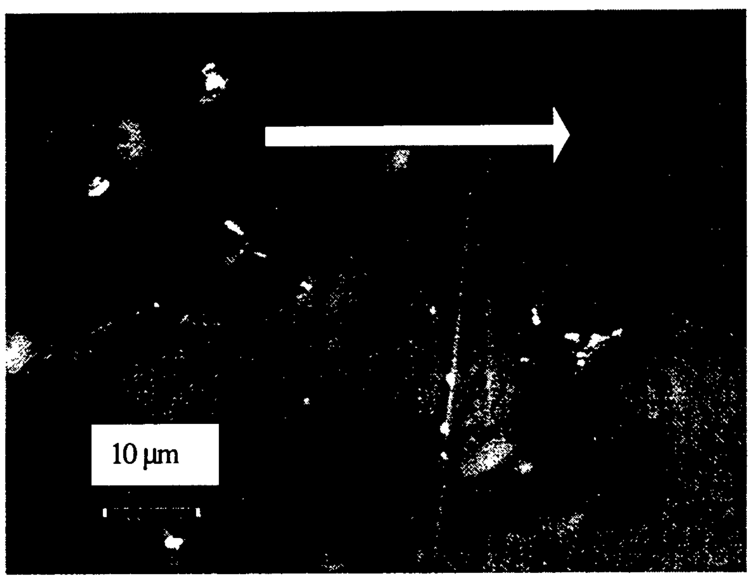


Figure 4 The two major crack discontinuities observed under the optical microscope from a DCB specimen surface
The arrows show the direction of the crack propagation

In the region further away from the crack tip, the crack path was continuous, apparently without any unbroken ligaments. The bridging nature in this region is believed to be mainly frictional.

Fracture Process Zone Characterization at Elevated Temperatures: Cyclic Loading Experiments Set-up

The effects of elevated temperature testing are presented in Figure 5. Initially, at room temperature, linear elastic behavior is observed, where negligible sliding occurs. However, as the temperature is increased to 600°C, while maintaining similar loading conditions, the formation of a hysteresis is observed. It is believed that this is due to the relaxation of the normal (clamping) forces on the bridging grains resulting from the reduction of the thermal elastic anisotropic effects present at room temperature. This results in lower threshold loads for the sliding of contact points. Upon additional increase of temperature to 800°C further relaxation of the normal forces is observed, resulting in the ratcheting behavior previously observed at room temperature.

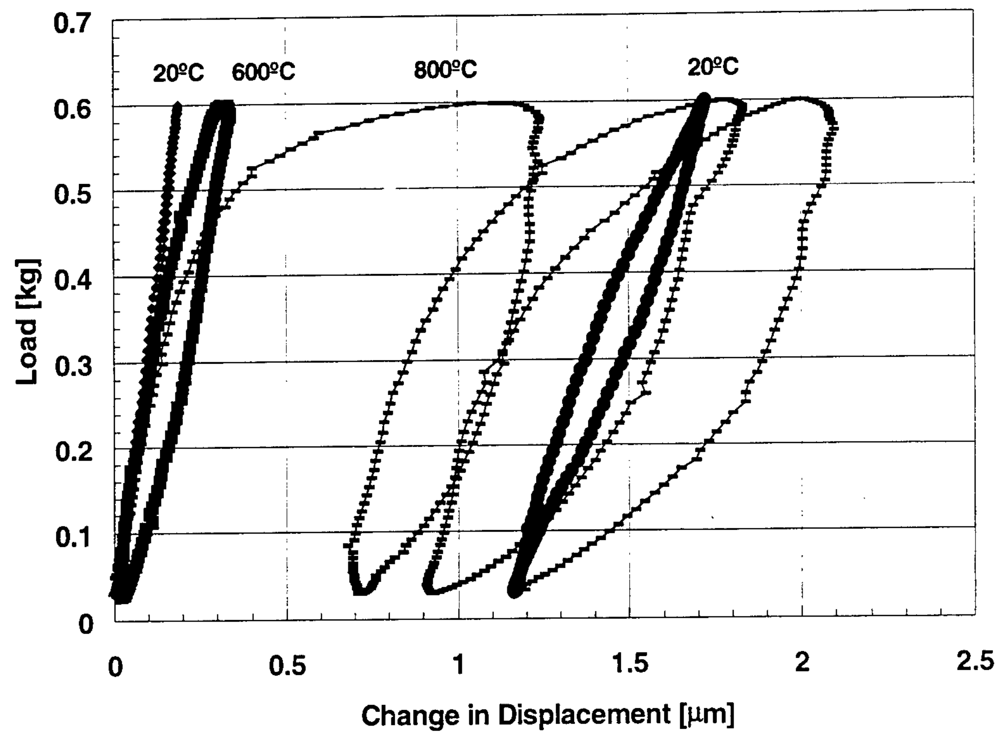


Figure 5 Effects of Temperature on PFT Behavior

As in the previous section, the comparison of the initial and final loading curves in this experiment shows evidence of FPZ degradation.

A comparison of the behavior of two alumina ceramics at elevated temperature is shown in Figure 6. This comparison is essential in the evaluation of the microstructural effects, since these curves correspond to initial crack opening displacements and temperature conditions that are similar. The initial crack opening displacements are estimated at 825 nm for both cases, where most of the grains are still active in the wake zone. The curves show that the load-displacement curve for AL23, which was tested at 0.45 Kg maximum load, would have probably failed at a slightly larger maximum load since the compliance at the maximum load point is very large.

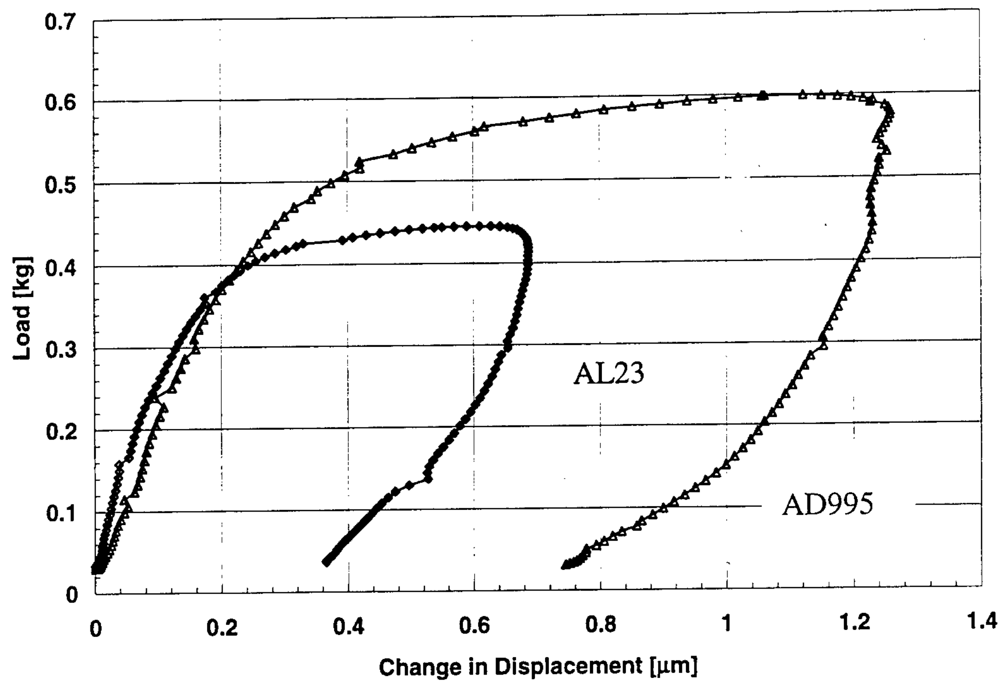


Figure 6 Elevated temperature (800 C) test loops comparing wake zone load capacity of a PFT 2 for two alumina microstructures

The AD995 curve, however, indicates a higher load or bridging capacity as it could be tested to 0.6Kg, with final compliance values that show a potential ability for higher load capacity. Considering that both PFT specimens were produced from temperature interrupted DCB fracture tests, and considering that the initial CODs and temperature conditions are similar, it could then be concluded that the differences observed in the load-displacement curves between the two alumina microstructures is strictly the result of active grain bridge systems. Since the initial crack opening displacements associated with these tests show that the small grain size range grouping (0-6 microns) for AL995 are active in the wake zone, then the additional load or bridging capacity for AD995 can, once again, be attributed to the ability of small size grains to provide load path between crack faces.

Micromechanical Modeling

An attempt to model the PFT by use of simplified modeling indicated very early in the program that the system, as modeled, would never have enough stored elastic energy to produce hysteretic loop behavior. It was clear from the initial results, that some distribution of elastic and frictional contributions was needed to adequately model cyclic loading behavior.

The incremental piecewise linear model is proposed as a representation of a set of wake zone elements groups that contribute to the total bridging event. The model, shown in Figure 7, represents the wake zone as a distribution of elements that have different elastic and slip characteristics. A distribution means that grain-bridging elements will have different degrees of contribution, i.e., some will move more easily than others will. The remainder of the grain bridges that do not contribute in slip or frictional dissipation, are modeled as the large spring (Elastic contact points and unbroken ligaments)

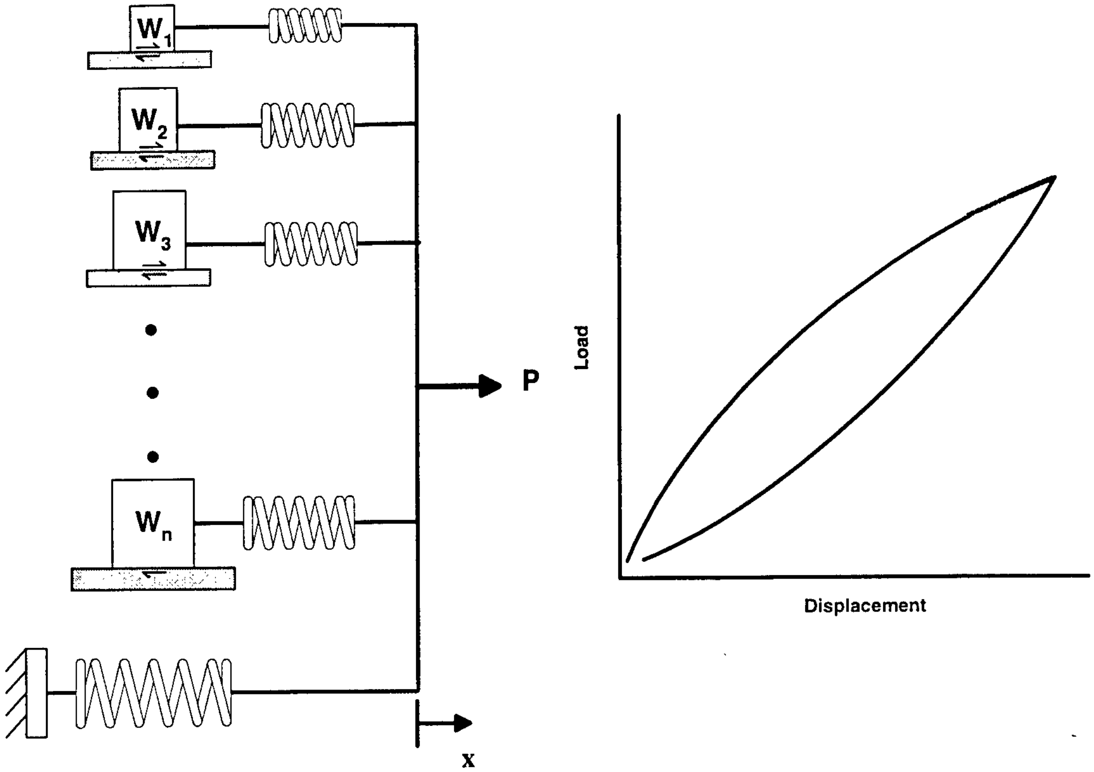


Figure 7 Schematic representation of the piecewise linear model and a resultant load-displacement record

Nearly closed-loop hysteretic behavior has been qualitatively described in the previous sections as the case where a high elastic contact point and unbroken ligament densities, are present in the wake zone. Shown in Figure , is a typical nearly-closed hysteresis loop corresponding to a AD995 PFT 1. Unlike the results of simplified modeling, a correlation to these test conditions was clearly obtained by use of the piece-wise incremental model.

To achieve this correlation, the modeling was subjected to the restrictions of a maximum initial compliance of 0.2 μm/Kg. A statistical distribution of normal forces and elastic contributors, as represented by the mass of the blocks and springs were obtained. These distributions were subject to the conditions that the elastic contributions could not exceed

52 N/ μm, which is equivalent to the initial stiffness of the system evaluated here. The individual elastic stiffnesses which comprised the distribution ranged between 2.3 and 5.1 N/ μm. An additional stiffness element of 5 N/ μm is added, which represent the remainder of elastic contributions that stay elastic throughout the simulation, i.e., do not contribute with sliding effects.

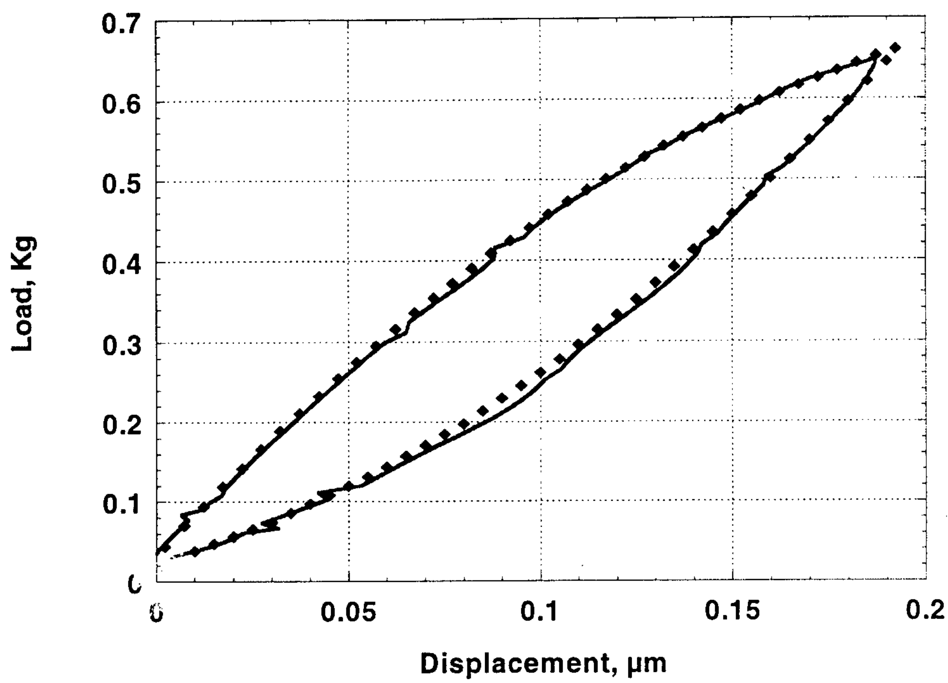


Figure 8 Room temperature incremental piecewise model correlation

The incremental piecewise modeling also provided excellent elevated temperature correlation. An example of the correlation of Thermal Expansion Anisotropic (TEA) effects is shown in Figure 9. In here, back-to-back tests are performed on the same specimen at similar test conditions. A room temperature test is first performed, which is then followed by an elevated temperature test at 600C. The test results are shown by the solid lines.

A successful correlation was obtained by first obtaining a correlation at room temperature. A correlation is then obtained at 600 C, by modifying the residual stress distribution array by reducing the normal forces by 42%, which is in excellent agreement with residual stress reduction calculations available in the literature.

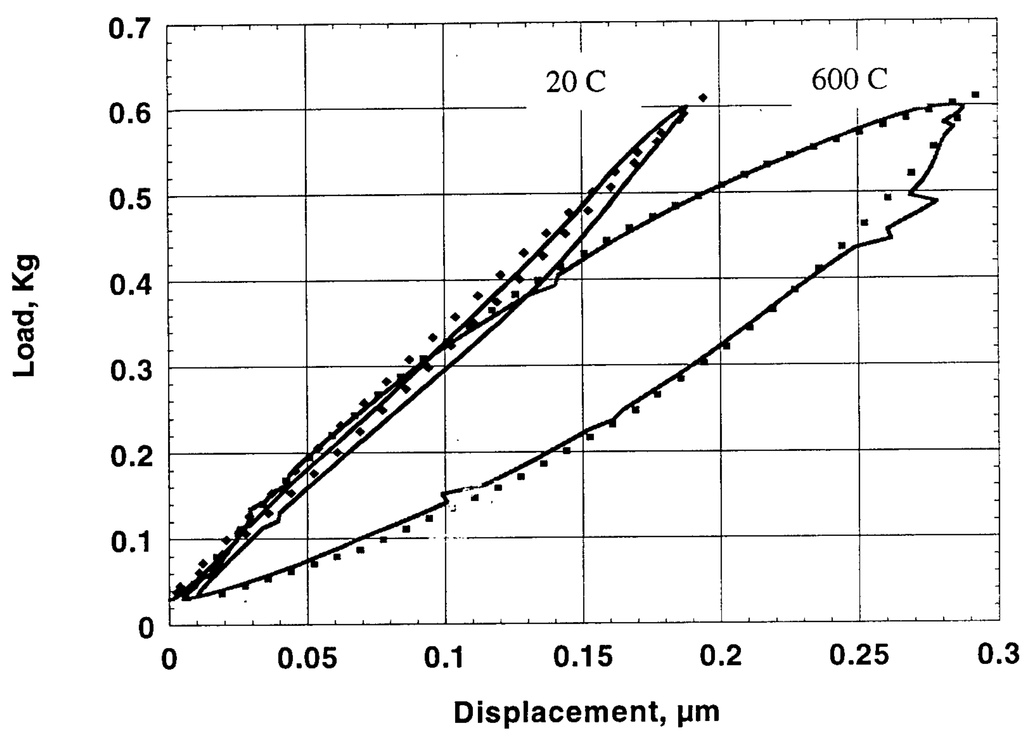


Figure 9 Correlation of thermal expansion anisotropic effects

An important finding of the correlation exercises is that there are microstructural events that can not be accurately modeled. These features include observations that tests performed on specimen of larger COD and that are associated with higher loads, causes the effective bridging lengths of smaller grains to be exceeded, causing dislodging and repositioning of these particles in the wake zone. The particles then interfere in the motion of the wake, causing artificial crack opening displacements, which are later reduced as the result of pulverization or accommodation of debris under subsequent cycles of loading. It is important to recognize that the scale of residual displacement falls in the order of this particle size, considering the cumulative residual displacement for the entire test sequence. An example of large-scale debris is shown in Figure 10.

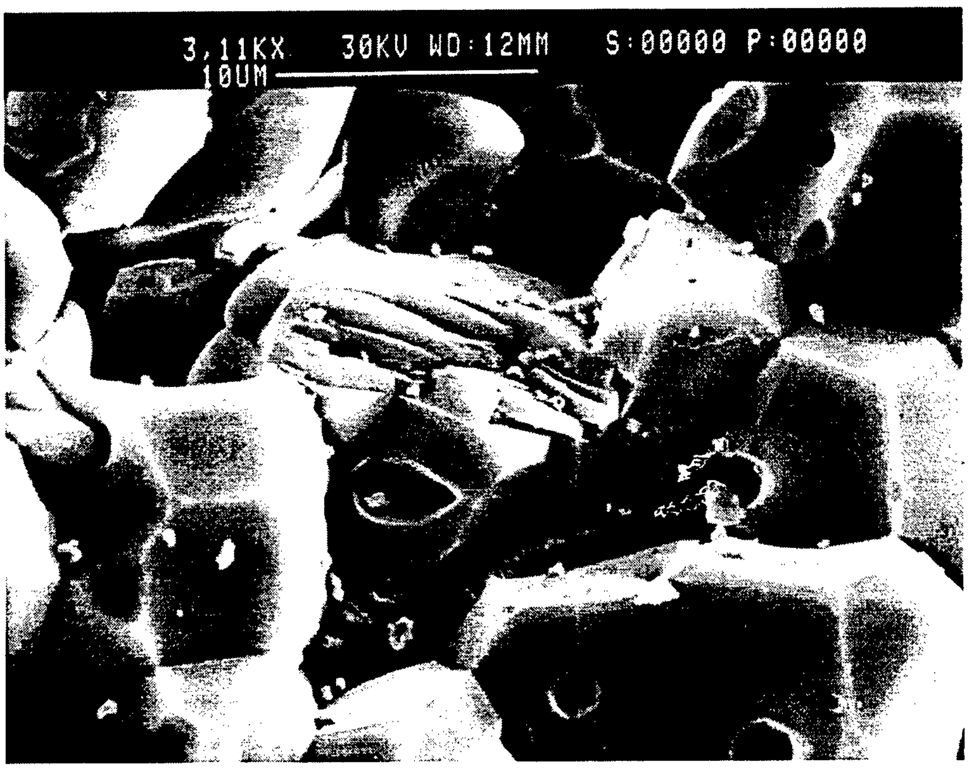


Figure 10 Fracture surface topography showing large scale debris in AD995

5. *Personnel Supported:*

Faculty: Ken W. White, Associate Prof., University of Houston

Graduate Students: Carlos R. Ortiz-Longo, University of Houston

6. Publications (Last 12 months)

- D.K. Tran, A.S. Kobayashi and K.W. White, "Crack Growth in Alumina at High Temperature," Engin. Frac. Mech., 68 149-161, (2001)
- F. Yu and K. W. White, "Relationship Between Microstructure and Mechanical Performance of a 70%Si3N4-30%BAS Self-Reinforced Ceramic Composite," *J. Am. Ceram. Soc.84 [1] 5-12 (2001)*
- Feng Yu, Nandakumar Nagarajan, Yi Fang, K. W. White, "Microstructural Control a 70% Si_3N_4 Barium Aluminum Silicate (BAS) Self-Reinforced Composite," *J. Am. Ceram. Soc.84 [1] 13-22, (2001)*
- T. Fett, D. Munz, R. D. Geraghty, K. W. White, "Bridging stress determination by evaluation of the R-curve," *European Ceramic Society*, **20** (2000) 2143-48.
- T. Fett, D. Munz, D. X. Dai, and K. W. White "Bridging Stress Relation from a Combined Evaluation of the R-curve and Post-fracture Tensile Tests," *Int. J. of Fracture,104, 375-85* (2000)
- Y. Fang, F. Yu and K. W. White, "Bimodal Microstructure in Si_3N_4-BAS Ceramic Matrix Composite by Pressureless Sintering," *J. Am. Ceram. Soc.* **83**[7] 1828-1830(2000)
- T. Fett, D. Munz, R. D. Geraghty, K. W. White, "Influence of specimen geometry and relative crack size on the R-curve," *Eng. Fract. Mech.* **66**, [4], 375-386 July 2000,
- M. Lu, J. W. Rablais, F. Yu and K. W. White, "Radiation Enhanced Diffusion of Ti in MgO," accepted for publication in *J. Appl. Phys.* (2000)
- M. T. Kokaly, D.K. Tran, A.S. Kobayashi, X. Dai, K. Patel, K.W. White, "Modeling of Grain Pullout Forces in Polycrystalline Alumina", Materials Science and Engineering – A285, 151-57 (2000).
- Y. Fang, F. Yu and K. W. White, "Microstructural Influence on the R-Curve Behavior of a 70% Si_3N_4- 30% Barium Aluminum Silicate Self-Reinforced Composite," *J. Mater. Sci.* **35** 2695-2699(2000)
- R. Geraghty, Hay, J.C., White, K.W. "Fatigue Degradation of Grain Bridging Elements in a Monolithic Alumina," Acta Metallurgica et Materialia , 47 [4], 1345-53, (1999)
- D.K. Tran, A.S. Kobayashi, K.W. White, "Process Zone of Polycrystalline Alumina", Experimental Mechanics., **39** [1], 20-24, (1999)

7. Interactions/Transitions:

- "Microstructural Design to Improve the Reliability of Pressureless Sintered Si_3N_4/BAS Composites," F. Yu and K. W. White, Paper No. S2-017-01 25th Annual International Conference on Advanced Ceramics and Composites, January 21-26, 2001, Cocoa Beach, FL

- "Microstructural Mechanisms of Cyclic Degradation in Structural Ceramics," ***(Invited)*** (IzfP) Fraunhofer Institute, Saarbrucken, Germany, Feb. 2000.

- "Improved Reliability of Pressureless-Sintered Si_3N_4/BAS Composite by Microstructural Modification," F. Yu, G. Himmler and K. W. White, Paper No. B2-029-00, 102nd Annual Meeting of the American Ceramic Society, April 30-May 3, 2000, St. Louis, MO.

- "Thermal Fatigue Test of Si_3N_4/BAS Composite," F. Yu, H. Fang, K. Ravi-Chandar and K. W. White, Paper N. B2P-013-00, 102nd Annual Meeting of the American Ceramic Society, April 30-May 3, 2000, St. Louis, MO.

- "Evaluation of the Microstructure-Interfacial Phenomena Relationship in the Fatigue Behavior of Monolithic Ceramics", C.R. Ortiz-Longo, K.W. White, 102nd Annual Meeting of the American Ceramic Society, St. Louis, MO, April 30-May 2, 2000.

- "Interfacial Issues in Fatigue of Monolithic Ceramics," R. Garaghty, K.W. White, 102nd Annual Meeting of the American Ceramic Society, St. Louis, MO, April 30-May 2, 2000.

- "Residual Stress Estimation in Alumina, Y. Fang, K. Ravi-Chandar, K.W. White, 102nd Annual Meeting of the American Ceramic Society, St. Louis, MO, April 30-May 2, 2000.

- "Effective Parameter in Load Transmission in Ceramic Structures," M. Doan, L. Wheeler, K.W. White, 102nd Annual Meeting of the American Ceramic Society, St. Louis, MO, April 30-May 2, 2000.

- "Interfacial Phenomena in the Fatigue Crack Growth Behavior of Monolithic Ceramics", C.R. Ortiz-Longo, R. Geragthy, L. Olasz, and K.W. White, 51st Pacific Coast Regional Meeting (PCRM) and the ACerS Basic Science and Electronics Divisions Meeting, Bellevue, WA, Oct. 27-29, 1999.

8. ***New Discovers:*** ***None***

9. ***Honors/Awards***

K.W. White
Faculty Research Award, Cullen College of Engineering, 1995

Editorial/Organizing Committee Member: Fracture Mechanics of Ceramics (Since July 1995)

5. ***AF Relevance***

- Develop microstructure-based methods for design of improved toughness ceramics.

- Initiate basis for component design methodology for brittle materials.